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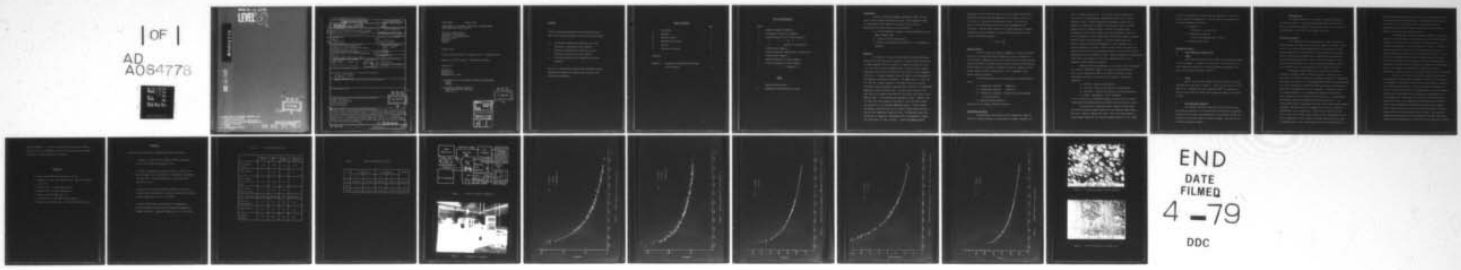

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LASER PULSE METHOD
HIGH TEMPERATURES

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Thermal diffusivity measurements from 500-2300K have been made on the following materials using the laser pulse method. (a) Two mutually perpendicular directions of a three dimensional carbon/carbon fibre composite (b) Parallel and perpendicular directions of a unidimensional carbon/carbon fibre composite (c) The matrix material used in fabricating the above composites. The use of a computer based digital data acquisition system significantly enhances the precision and accuracy of the diffusivity measurements.

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# MEASUREMENT OF THE THERMAL DIFFUSIVITY OF CARBON/CARBON FIBRE COMPOSITES FROM 20-3000°C.

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November 1978

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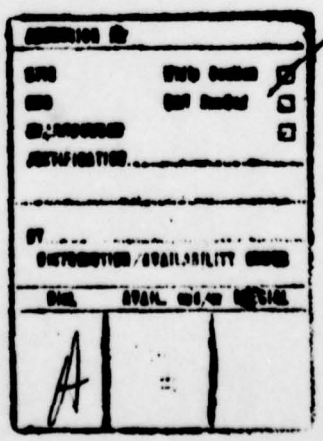

ABSTRACT

Thermal diffusivity measurements from 500-2300K have been made on the following materials using the laser pulse method:

a) Two mutually perpendicular directions of a three dimensional carbon/carbon fibre composite

b) Parellel and perpendicular directions of a uni dimensional carbon/carbon fibre composite

c) The matrix material used in fabricating the above composites

The use of a computer based digital data acquisition system significantly enhances the precision and accuracy of the diffusivity measurements.

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## Introduction

The pulse diffusivity apparatus developed at UMIST has been used to obtain thermal diffusivity data over a wide temperature range on carbon/carbon fibre composites of interest to the U.S.A.F.. Measurements have been carried out from 500-2250K on

a) A three dimensional composite in directions parallel to the three orthgonal axes.

b) A sample of matrix material only.

c) Parallel and perpendicular directions of a one dimensional composite.

## Apparatus

The theory of the pulse or flash method for measuring thermal diffusivity is sufficiently well known not to warrant further description. The UMIST apparatus has been outlined in a number of publications (e.g. 1, 21) and will be briefly summarised here. The facility has a design capability from 300-3300K for operation under vacuum or inert atmosphere conditions. A schematic diagram is given in figure 1 and a photograph of the apparatus is presented in figure 2. The sample which is in the form of a thin disc having a diameter in the range 6.35-10 mm is mounted horizontally inside a graphite susceptor which is heated to the measurement temperature using an induction coil. The heat pulse incident on the upper face of the sample is provided by a solid state ruby laser of 100 J maximum output. Pulse durations are typically 0.8 msec. Radiation from the lower face of the sample is collected by a lens and mirror system and focussed on to an infra-red temperature sensor. Suitably biased, the change in resistance per unit of incident radiant energy provides a record of the temperature change with time. The amplified output from the detector is logged by a minicomputer which is programmed to sample 1024 data points at 1 msec intervals. Suitable programming permits

computation of a half rise time value ($t_{\frac{1}{2}}$) to a precision better than 1 microsecond, and the transient amplitude ratio at either 5 $t_{\frac{1}{2}}$ or 10 $t_{\frac{1}{2}}$ to that at $t_{\frac{1}{2}}$. Using one of these ratios the analysis outlined by Cowan (3) for correcting heat losses has been programmed and $w/\pi^2$ calculated. Finally length correction for thermal expansion is applied automatically and the computer calculates thermal diffusivity $\alpha$ using the three determined parameters

$$\alpha = (w/\pi^2) \frac{L^2}{t_{\frac{1}{2}}}$$

## Material Details

The basic aim of the research programme is to model the thermal properties of carbon fibre/carbon composites in terms of properties of the constituent components, such as fibre conductivity and volume fraction, matrix conductivity and volume fraction and pore content and distribution. To that extent it is desirable to measure at least two composites and if possible matrix and fibre material alone. Such a programme should improve predictive ability.

The following materials have been supplied by Wright-Patterson A.F.B.:

1) 3-dimensional composite COMPOSITE A
2) 3-dimensional composite COMPOSITE B
3) 1-dimensional composite. Unidirectional fibres embedded in matrix material.
4) Graphitised matrix material.

Details of all the samples are given in table 1.

## Experimental Procedure

Although results are required over the temperature range 300-3000K it is neither possible nor practicable to attempt to generate the

data in a single continuous run. In the first instance temperatures below 1500K are recorded using a thermocouple; above 1500K an optical pyrometer is used. Elevated temperature measurements require that the apparatus be disassembled to remove the thermocouple, align an optical pyrometer and fit a smaller susceptor insulated with graphite felt. Furthermore the PbS detector normally used for measurements >500K has a cut-off wavelength at 3 μm so measurements from 300-500K require a detector having a longer cut-off wavelength. A liquid nitrogen cooled InSb detector having a detectivity extending to 5.5 μm is available but a suitable amplifier was lacking. Effort has been devoted to building this amplifier which has now been successfully tested. For this reason however measurements have only commenced at 500K. Finally measurements at temperatures >2300K need to be carried out in an inert atmosphere because of the significant vapour pressure of graphite.

Bearing in mind the foregoing it is convenient to make measurements in a series of temperature ranges on a series of samples rather than over the whole temperature range on one sample or material, thereby conditioning the order in which results are obtained. These may be summarised:

1) 300-700K Using the InSb detector.

2) 500-1500K. Using the PbS detector and thermocouple.

3) 1200-2300K. Using the PbS detector and optical pyrometer.

4) >2000K. Measurements in inert atmosphere.

It will be noted that a considerable degree of overlap has been allowed thereby permitting identification and elimination of any relative errors.

The procedure for sample production consists of sectioning a slab from the material supplied thicker than the expected sample length. From this a number of samples were cored. After trial measurements an optimum sample length was calculated and samples machined to this length.

At least two specimens will be measured per orientation to allow for possible material inhomogeneities. Present effort has concentrated on obtaining measurements as follows.

1) Composite A:

Parallel to Z, X and Y axis.

2) Unidirectional Composite

Parallel and perpendicular to fibres.

3) Matrix material alone.

## Experimental Results

### A. Three dimensional Composite "A"

#### 1) Z-axis

The thermal diffusivity from 500-2300K of two separate specimens is shown in figure 3. Scatter in the results is less than 3% over the whole temperature range and the results show excellent agreement with data on this material supplied by AFML.[4]

#### 2) X-axis

Results for two X-axis samples from 500-2300K taken over three measurement runs are given in figure 4. Scatter in the results is of the order of 5%. Agreement with data supplied by AFML[4] is excellent at 500K and above 1200K but between these temperatures AFML results deviate by 7-8%.

### B. Uni-dimensional composite

Two specimens have been tested from 500-2300K one for which the fibres were parallel to the specimen axis and the other a transverse specimen in which fibres are perpendicular to the direction of heat flow. Results for the parallel sample are given in figure 5 and those for the transverse specimen in figure 6.

C. Matrix Material

The thermal diffusivity of a sample of matrix material alone is plotted in figure 7. However detailed examination of this material reveals a high porosity material (∿40%) having a density of ∿1.35 gm $cm^{-3}$. The average pore size (fig. 8) lies between 100-300 μm.

Discussion of Results

The z direction of the three-dimensional composite exhibits a diffusivity some 35% higher than that of the samples cut parallel to the X axis. Since fibres oriented perpendicular to the direction of heat flow are expected to be less efficient in conducting heat than parallel fibres these results are entirely self consistent with the observation that the fibre volume fracture in the Z axis is 0.22 whereas in the X-axis the fraction is only 13%. Clearly, however the diffusivity ratios do not exhibit a one to one correlation with the fibre volume ratio.

Pending more detailed analysis of the results it would be premature to attempt to draw too many conclusions. However the data published on the thermal conductivity of pyrolytic graphite (5) demonstrates conclusively the large ($2 \times 10^2$) anisotropybetween parallel and perpendicular directions. Anisotropy in conductivity between parallel and perpendicular directions of a graphite fibre is to be expected. Consequently values of the thermal diffusivity of a unidimensional composite should provide useful information on the diffusivity of fibres alone. Such experimental procedure has already been demonstrated by Lee and Taylor (6). The thermal diffusivity of the parallel direction of the unidirectional composite are greater than those of the 3-D composite Z and X axes by factors of approximately 3 and 4 respectively. Again this is broadly in accord with the fact that the fibre volume fraction of the 1-D composite is 0.53. However perpendicular to the fibres the thermal diffusivity is one order of magnitude lower. This is much smaller than the anisotropy ratio of

highly oriented graphite and clearly reflects the contribution from the 47% volume fraction of matrix material. Scanning electron microscopy views figure 9 tend to reinforce this opinion since the lack of separation between fibres and matrix strongly indicates that matrix material constitutes a well defined heat path.

The diffusivity of the matrix material exhibits a diffusivity higher than the diffusivity of either direction of the three dimensional composite which substantiates the opinion that matrix material can contribute significantly to heat conduction in a composite. However the high porosity and low density is not strictly representative of the composite matrix which exhibits a much lower porosity (figure 9). This may reflect unavoidable differences in processing conditions whereby fibre/matrix interfaces in the composite may facilitate migration and evaporation of voids which would otherwise be trapped in the graphitised matrix. This suggests circumspection in attempting to define a diffusivity or conductivity value to the matrix component of the composite.

Taylor et al (7) have shown that the thermal conductivity of a polycrystalline graphite may be expressed in terms of the conductivity of the graphite layer planes reduced a factor which combines components due to porosity and tortuosity. Hence the conductivities/temperature curves for different directions in a polycrystalline anisotropic graphite artefact may be superimposed by multiplying by an appropriate factor. If we attempt such a comparison for our data then by normalising the diffusivity values at one temperature and determining the diffusivity ratios at other temperatures the diffusivity curves may be compared. Table 2 shows values for the five sets of data normalised at 1000K and compared with the values obtained at 500K and 2000K. The normalised diffusivity curves for the X and Z directions of the three dimensional composite and the perpendicular direction of the one dimensional composite

show good agreement. In contrast the matrix material and the parallel direction of the one dimensional composite also show excellent agreement but exhibit a stronger temperature dependence.

## References


1. R. Taylor (1972) High Temp.-High Press., 4, 649.
2. J. Ormerod, R. Taylor, R.J. Edwards (1978). Metals Technology 5 (4), 109.
3. R.D. Cowan (1963) J. Appl.Phys., 34, 926.
4. S. Theibert (1977) private communication.
5. R. Taylor (1966) Phil.Mag., 13, 157.
6. R.E. Taylor and H.J. Lee (1975) Carbon, 13, 521.
7. R. Taylor, K.E. Gilchrist and L.J. Poston (1968) Carbon, 6, 537.

## Appendix 1

Publications and Presentations completed during current contract.

1. J. Ormerod, R. Taylor and R.J. Edwards "Thermal diffusivity of cast iron", Metals Technology 5(4) 109.

2. R. Taylor, "Thermophysical property research on solids in the United Kingdom", Proc.7th Symposium on Thermophysical Properties, 10-12 May 1977. Gaithersburg, Maryland, Am.Soc.Mech.Eng., New York, p.37-42.

3. R. Taylor and C.M. Fowler, "Thermal diffusivity of pure iron and dilute iron alloys", Proc. XV Thermal Conductivity Conference, Ottawa, Canada, Aug. 24-26 1977 (In Print).

4. R. Taylor, "Metallurgical Applications of Thermophysical Property Research",(invited paper)VI European Thermophysical Property Conference. Dubrovnik, Yugoslavia, 26-30 June (1978).

Table 1. Materials Specification

| | Composite "A" | Composite "B" | Matrix Graphite | 1-Dimensional Composite |
|---|---|---|---|---|
| Bulk density $g.cm^{-3}$ | 1.883 | 1.917 | 1.36 | 1.915 |
| Open porosity % | 6.1 | 5.95 | - | - |
| Fibre volume fraction X and Y axes | 0.13 | 0.139 | N/A | - |
| Fibre volume fraction Z axis | 0.22 | 0.132 | N/A | - |
| Fibre cross-section | Crenulated | Crenulated | N/A | Crenulated |
| Fibre bulk density $g\ cm^{-3}$ | 1.66 | 1.66 | N/A | 1.66 |
| Fibre diameter µm | 6.50 | 6.50 | N/A | 6.50 |
| Filaments per yarn | 1440 | 1440 | N/A | 1440 |

Table 2. Normalised Thermal Diffusivity

| T.K. | 3-D Composite | | 1-D Composite | | Matrix |
|---|---|---|---|---|---|
| | X | Z | $\parallel$ | $\perp$ | |
| 500 | 1.69±0.05 | 1.70±0.05 | 1.87±0.05 | 1.70±0.05 | 1.87±0.05 |
| 1000 | 1 | 1 | 1 | 1 | 1 |
| 2000 | 0.65±0.03 | 0.66±0.03 | 0.57±0.03 | 0.64±0.03 | 0.56±0.03 |

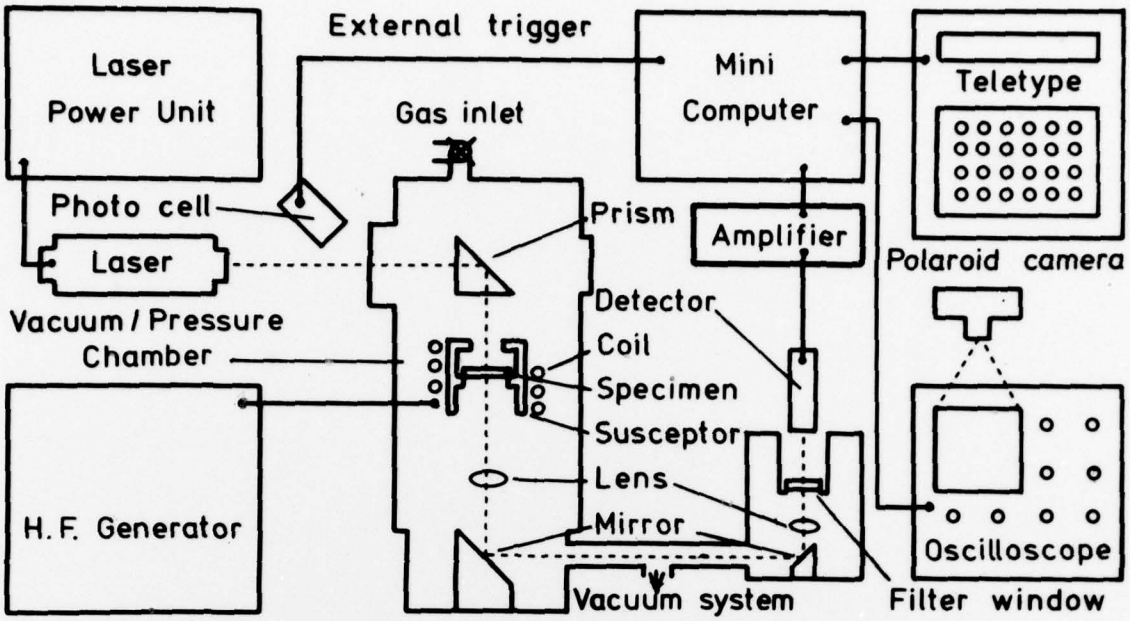


Figure 1. Schematic diagram of apparatus.

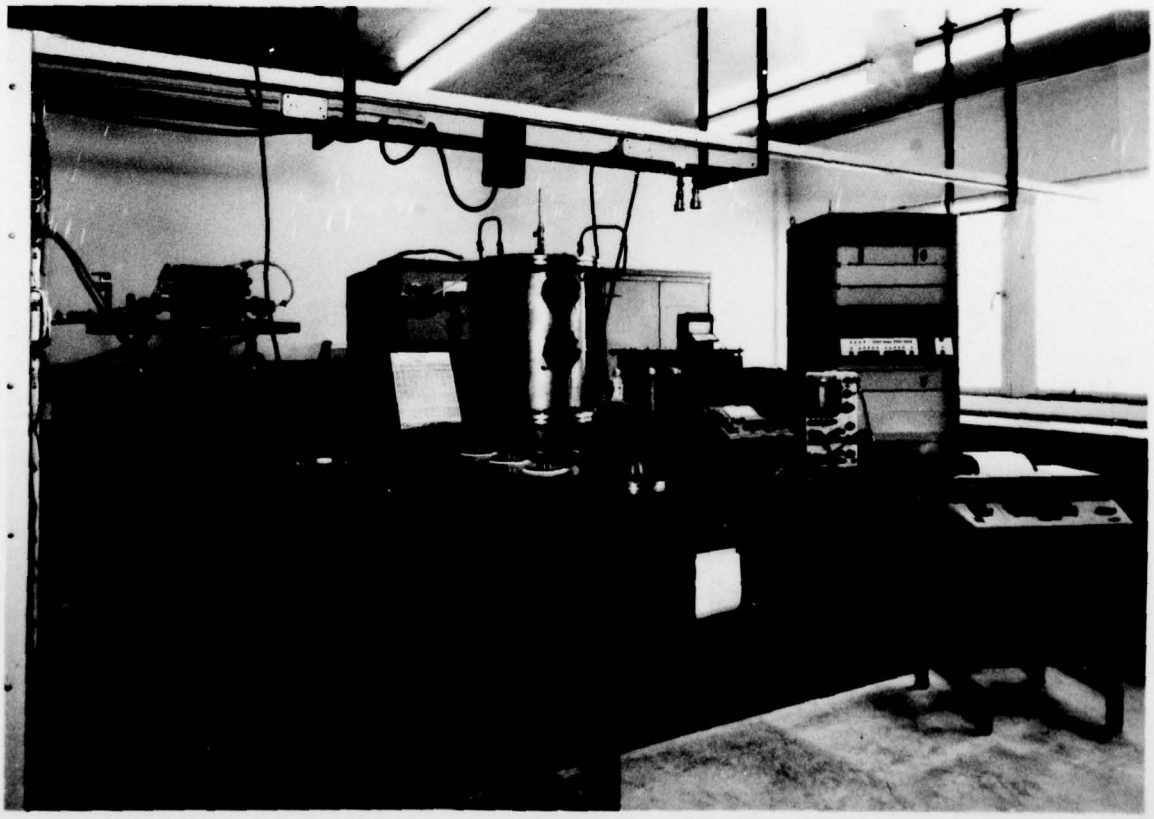

Figure 2. Photograph of equipment.

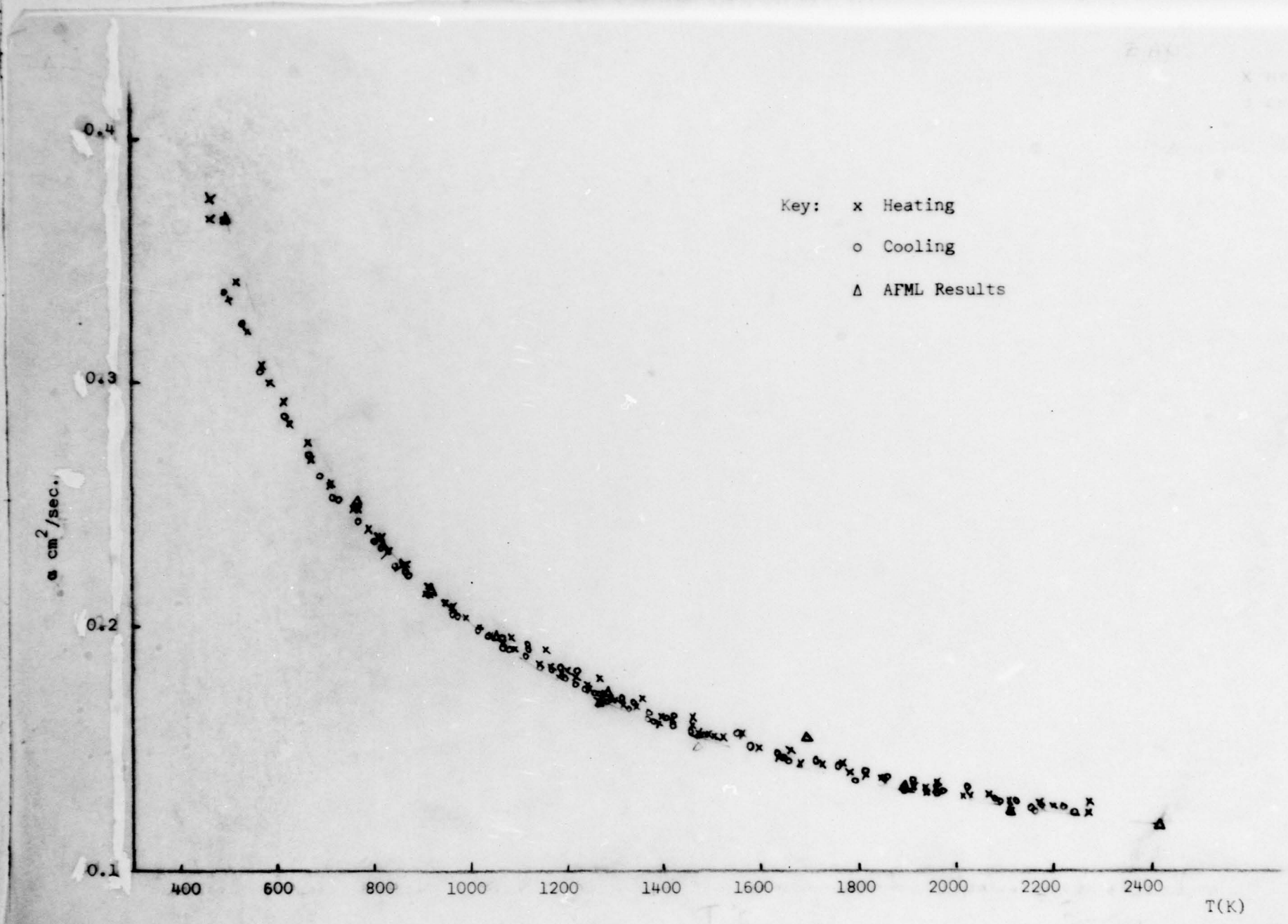


Figure 3. Thermal diffusivity of 'Z' axis composite A.

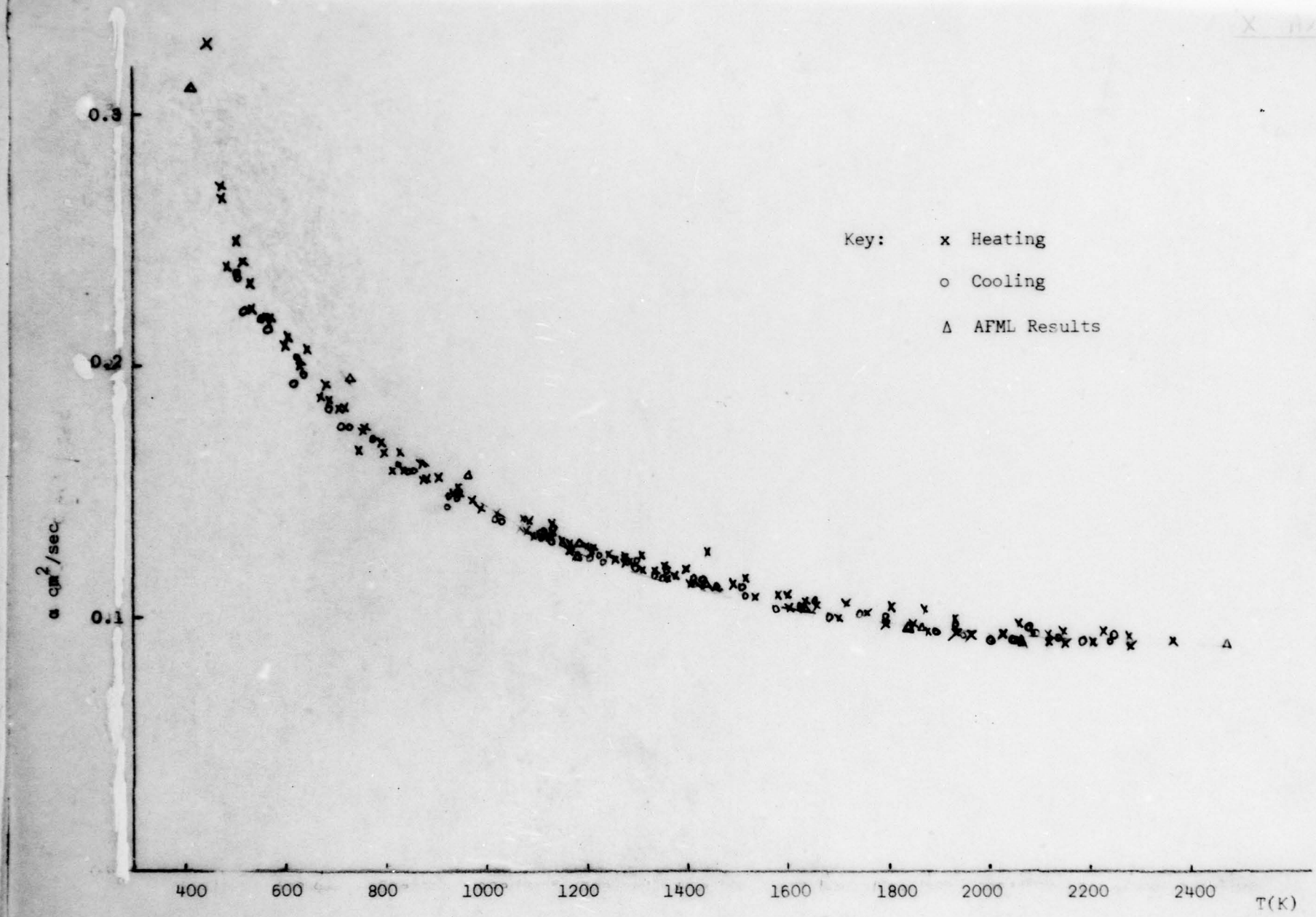


Figure 4. Thermal diffusivity of 'X' axis composite A.

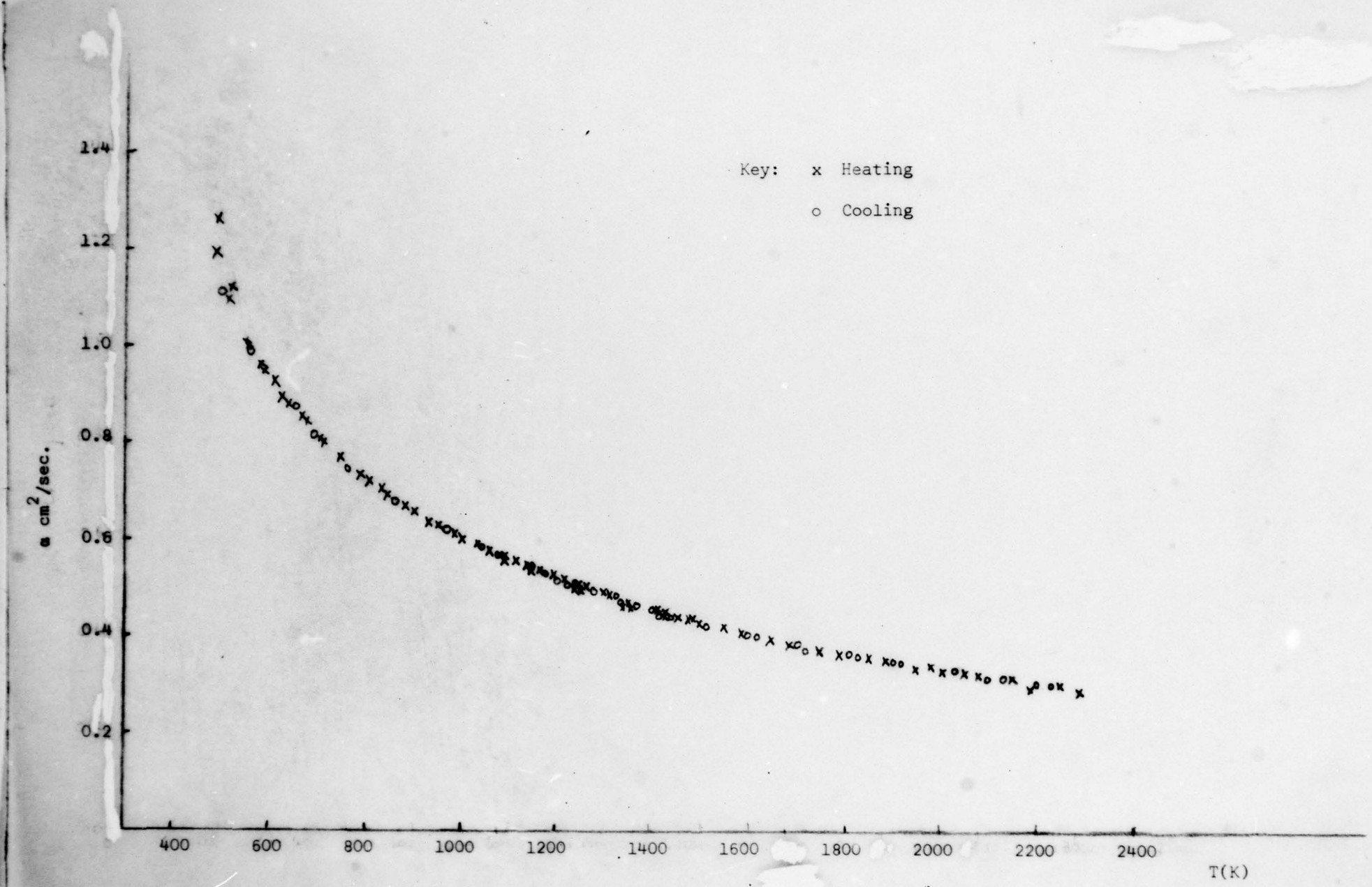


Figure 5. Thermal diffusivity parallel to fibre axis of unidirectional composite.

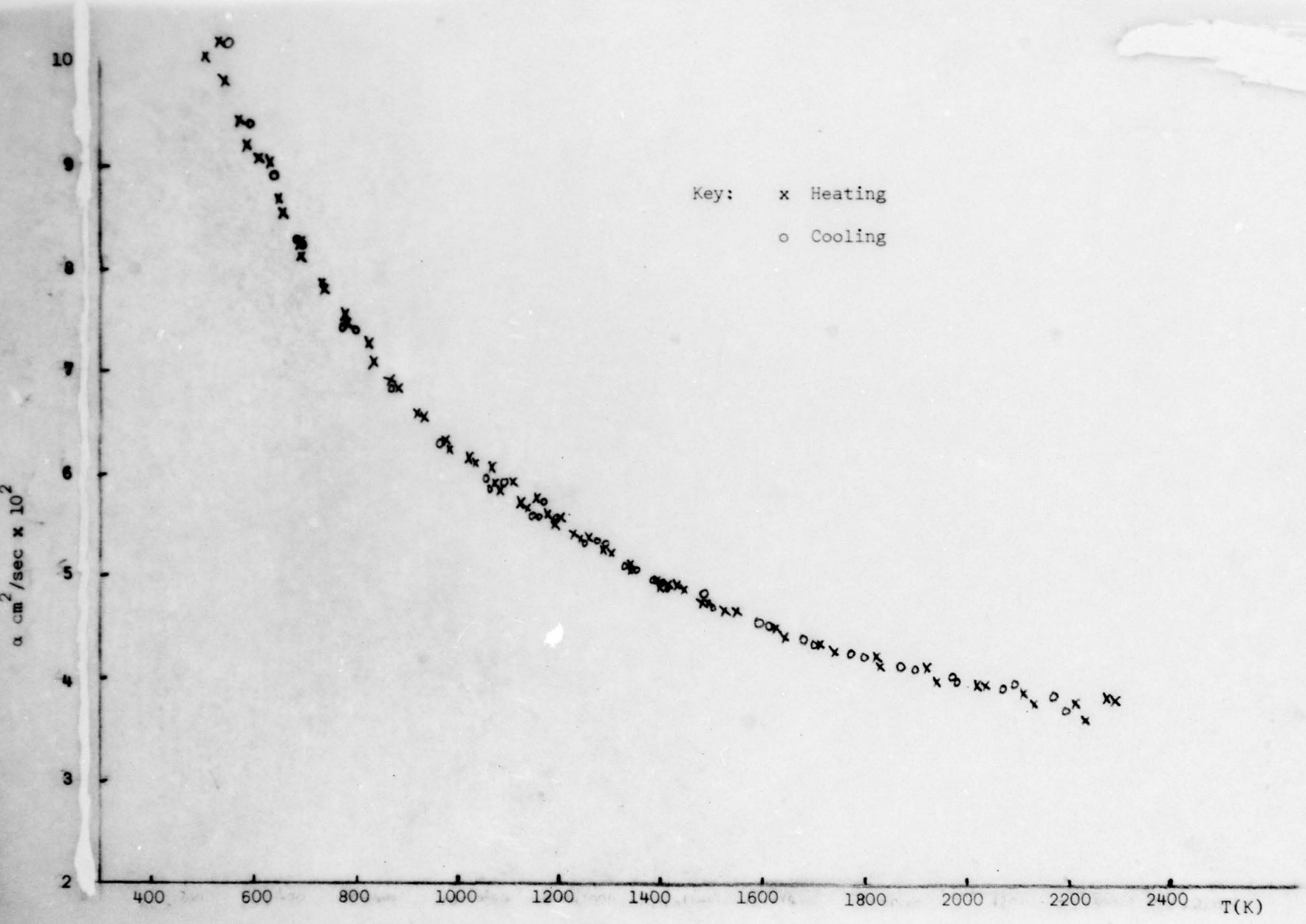


Figure 6. Thermal diffusivity perpendicular to fibre axis of unidirectional composite.

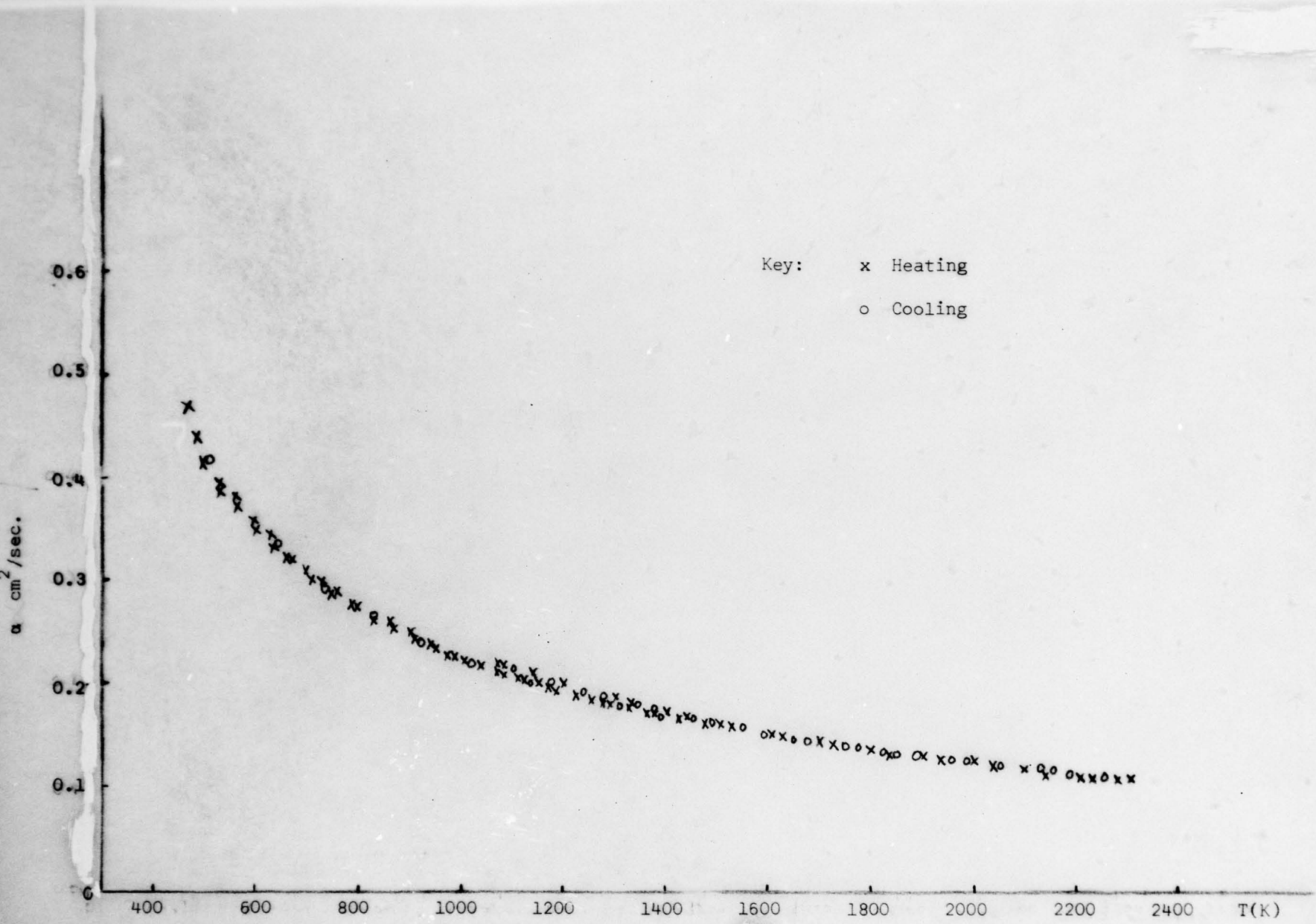


Figure 7. Thermal diffusivity of matrix graphite.

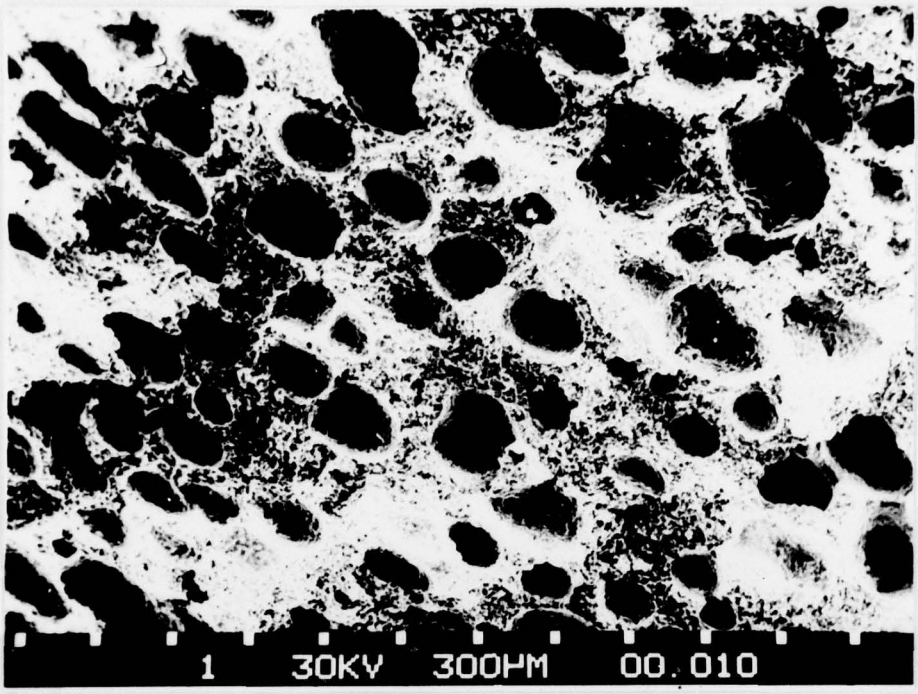


Figure 8. Electron micrograph of matrix graphite.

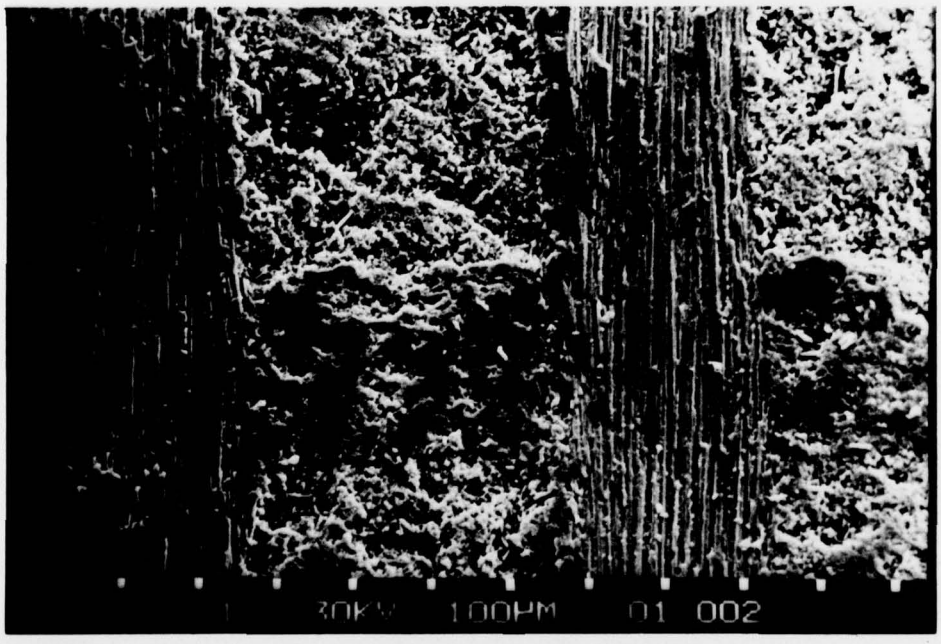


Figure 9. Electron micrograph of composite "A".